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Insights beside assessments into small volume cyclic response of elastic plastic systems

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Abstract

In contrast to monotonic load studies, the engagement with a cyclic time dependent processes of small volume material response has been restrained. Crack tolerance in thin films almost by definition is restricted. Nevertheless, in terms of structural stability the fatigue resistance remains a critical concern regardless the scale. The cyclic displacement amplitudes can be enhanced by remote mechanical stress/strain or thermal origins resulted in similar outcome of cumulative irreversible micro-plasticity damage. Thus, the cyclic life in nano applications is mainly dominated by the crack nucleation controlled process. The current study is centered on the fatigue crack initiation stage as a first order design pillar and therefore require additional experimental input. Activities in elastic-plastic polycrystalline and single crystal systems in various crystal structures are described. Beside findings regarding the fatigue cracks initiation life, the role of compressive residual stress on the cyclic response has been also analyzed. The fatigue crack initiation criteria have been associated to other constitutive assessments that are related mainly to the crack stability equation elements. In addition, completely different insights are introduced into the concept of the critical film thickness, indicating the susceptibility to the crack formation. As addressed in heteroepitaxial layers, specific analogous characterization exists either in dislocation activities or by triggering crack formation. The physical aspects of threading dislocations growth and glide behavior and local decohesion ramifications are highly dependent on the layer thickness. Experimentally based, under strain control conditions at ambient temperatures the aforementioned fatigue crack initiation stage in small volume segments, obeyed a cumulative damage mechanism. It was also established that the low energy dislocation structures and the initiation life were dramatically dependent on the strain amplitude range. The role of compressive residual stress in prolonging the fatigue initiation life was also consistent. Even in silicon based materials, dislocation activities prevailed. It became apparent that categorical generalization in nano-silicon thin film fatigue behavior is beyond the susceptibility to the native oxide or environmental interaction. Issues like film processing methodology, film/substrate interfaces, residual stresses and micro-cracking feasibility occurrence as wall as critical sites still require further design insights.

Keywords: fatigue; elastic-plastic systems; nano-scale; residuals; initiation; design

1. Introduction

It appears that from a practical point of view (mainly due to geometrical constraint) the cyclic life in small volume applications is dominated by a crack nucleation-controlled process. Thus, two-fold major research activities emerged. First, to intensify efforts in order to gather additional input into the issue of fatigue crack-initiation life and

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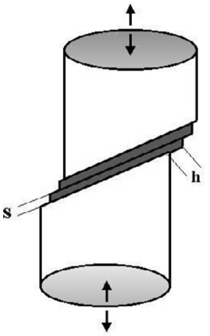
secondly to explore complex means in order to achieve a certain degree of crack tolerance regime. The persistent and consistent research drives into the latter become understandable in order to allow significant progress of the fatigue response or to prolong the total cyclic service life. Nevertheless, at the present juncture, desired activities like multi-layer segment advancement remain a long-term challenge, leaving critical event of the fatigue crack-nucleation stage to be further elaborated. In this context, various micro-mechanical sequential events have been proposed regarding the complex fatigue crack initiation occurrence [1-11]. Only briefly, one model attributes the onset of crack initiation to the gradual increase of the surface slip upset with the numbers of cycle dependence. Physically, the fatigue conditions impose partial irreversible micro-plasticity displacement attributed as such to the damage evolution. In addition, Mugrabi et al. [4] have stretched the surface roughening up to the development of slip localization that can lead to crack formation. Secondly, dislocation dynamic based models [8,10] that attributed local energetical instability, namely, strain energy accumulation with an end result to relaxation manifested by crack nucleation. Although not completely analogous to a small volume segment situation, the current first order approximation has been centered on near surface ill-defined layer by tracking nano geometrical events. Experimentally, this section has been performed in pure polycrystalline copper affected by cyclic strain. The aforementioned procedure has been assisted by micro-probe visualization and fine feature measurements that included also the introduction of residual stresses by mechanical shot peenning. Notice that in contrast to monotonic load studies, the engagement with mechanical time-dependent process in small volume application has been limited. Still, in thin films the fatigue process is highly relevant regardless the scale. Here, fatigue might involve mainly the crack nucleation stage in contrast to a possible and even desired sub-critical crack growth. The cyclic stress/strain field origin if by mechanically remote load/displacement or by thermal cycles become insignificant. Topics like the stress state and multi-axial effects are directly connected to the fatigue path beside the establishment of the fracture classification in thin films. Mechanical response in small volume segments differ from the known macro elements. Based on various considerations, frequently even dependent, film thickness reduction or the upper bounded strain/stress controlled design become dominants regarding the structural integrity evaluations. In this context, basic argumentations have been developed already in heteroepitaxial films demonstrating the critical cracking thickness concept [12-14]. In order to gain better resistance to failure, other insights emerged also from the strain energy release rate criteria [15,16]. Based on elastic foundation, the strain energy release rate is a function of the stress/strain field and the film thickness. The mentioned dominant variables have been explored from different approaches and they are elaborated in the present investigation. This is beside the notion that particularly in small volume cases broad generalization is highly restricted. Issues like film processing methodology, film/substrate interfaces, and residual stresses effects all are influential factors. In addition, the potential micro-cracking occurrence and their specific sites are considered here as decisive weakest links in terms of structural integrity.

2. Materials and experimental procedures

Fatigue model material, namely a face centered cubic polycrystalline commercially pure copper has been selected. Standard mechanical properties on the background of micro-structural characterization have been followed by fatigue tests. Cyclic tests at ambient temperature were conducted on uniform specimens under tension/compression strain controlled conditions. The uniaxial and uniform specimens consisted of cylindrical 6mm in diameter and a gage length of 25 mm. Only for the fatigue tests specimen, very limited area was flatted to allow fine scale surface feature observations. The specimen's preparation for monotonic and cyclic conditions included mechanical (0.25 μm) and electrolytical polishing, down to a nano-scale quality. Tension/compression cyclic tests for R=-1 and $5H_Z$ were performed in plastic strain range of 10^{-4} to 10^{-2}. In addition, three point bend specimens were utilized on electro-magnetic resonance set-up and crack initiation was tracked by the frequency reduction or by in-situ variations of the material compliance. In contrast, for the uniform specimens, the crack initiation was tracked by one step replication technique. Besides micro-crack initiation tracking, prior sequential events in terms of slip upset that formed at the external surface have been observed and measured by Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). The replication and the fine scale feature observation procedures have been applied in a step-wise periodic data collection along the monotonic increase of the fatigue cycle numbers. In addition, low energy dislocation structures were observed by Transmission Electron Microscopy (TEM) assisted also by images and by selected area diffraction at 200 KV. In a comparative approach the program was stretched also to a comprehensive residual stresses (RS) investigation. The insertion of RS can be achieved by various procedures and two of them have been presently utilized. First, by sharp thermal gradients and second, by surface modification due to shot peenning. In fact, both have been currently used in copper at RS levels of 0.4 to 0.7 of the materials yield stress. The insertion of RS and its intensity have been verified by X-ray diffraction methodology. The RS effects tracking were bounded either by the crack initiation life of by a given plastic accumulation degree in various strain amplitude values.



$$\sigma_{12} = \delta_{11}\delta_{12}\sigma_{11}$$

$$N \cdot f \cdot \Delta\varepsilon_p = \frac{h}{s}$$

where N the number of cycles and f the fatigue efficiency factor or

$$f = \frac{h'}{N \cdot \Delta\varepsilon_p s}$$

Fig. 1. (Left) Schematic, cyclic segment enhancing irreversible slip. (Right) Results shear test and fatigue efficiency formulation

Following the current strain-controlled cyclic amplitudes, dislocation activity resulted in slip-upset or that irreversible micro plasticity occurred that was locally measurable by AFM. Both the slip displacement (h) and the slip spacing (s) are shown in Fig. 1. This local experimentally-based approach enabled to conduct quantitatively a comparative study between specimens with or with no RS. By considering again the near surface slip-upset, the independent variable remains the applied strain amplitude affecting the surface roughness. Clearly, the measured slip-upset is still the consequence of only a small fraction of the dislocation population, the fraction that emerged at the free surface. The advancement in local displacement measurements proposes in fact a geometrical model for initiation in terms of the "fatigue efficiency" and this after defining the local strain as the ratio of h/s (see Fig.1). Crack initiation life vs. plastic strain amplitude is depicted in Fig. 2.

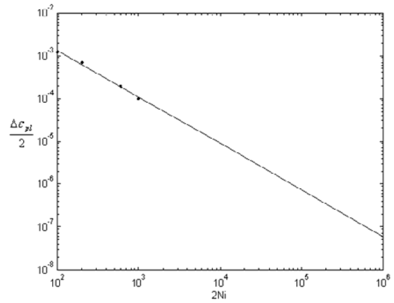


Fig. 2. Plastic strain amplitude vs. crack initiation cycles

Table. 1. Values determined by the current geometrical approach (*Ref. Harvey et al. – Acta Metall Mater, 1994)

Material/S.mod.	Strain amplitude	Efficiency %
Cu-NR	$5.3 \cdot 10^{-2}$	3.1
Cu-R	$2.2 \cdot 10^{-2}$	1.3
Cu-NR	$1.5 \cdot 10^{-3}$	2.1
Cu-R	$1.5 \cdot 10^{-3}$	1.7
HSLA*	$4.0 \cdot 10^{-4}$	0.9
αTi*	$4.0 \cdot 10^{-4}$	1.9

A comparison between typical values of the current defined "efficiency" with and with no RS are given in Table 1. As revealed, RS affect the fatigue efficiency for initiation. The efficiency as such is cycle-dependent influencing directly the fatigue ductility exponent. Notice that the fatigue initiation stage behaved also according to the damage mechanics description as addressed by the Manson-Coffin rule. However, for initiation stronger propensity has been manifested between the damage and the strain amplitude. For initiation the fatigue ductility exponent is approaching -1 in contrast to -0.5 normally proposed for the fatigue total life. In applications that are dominated by initiation controlled process (brittle or small volume layers) life dependency on the strain amplitude turns to be profound. Again, in contrast to monotonic load studies the engagement with time-dependent condition in small volume segments have been more restricted. Presently, the "fatigue efficiency" parameter has been selected as appropriate to assess cyclic sensitivity in terms of the effective cumulative damage. Here to mention that the importance of the fatigue process regardless the scale has been confirmed in nano-silicon particles indicating that fatigue resistance in small volume application remains essential [17].

3. Results, discussion and additional factors for design assessments

It seems noteworthy to emphasize similar conclusions that result from two completely different model bases, namely, geometry vs. energetics [8,10,11], regarding the cyclic initiation life. Specifically, both, Mura's school and the slip upset model, have been founded on dislocations dynamic. Beside model developments, virtual simulations in both became possible and brief comparison is given in Table 2. It appears that by following initially different approaches do not explore new avenues on the contrary, mutual confirmation is really established. However, the strong agreement in terms of critical variable trends, manifest again the dominant role of dislocations affecting the cyclic response. A completely different design factor to be assessed is the film thickness. Considering an elastic media the strain energy release rate G is given by [15, 16]:

$$G = g(\alpha,\beta)\frac{\pi(1-\nu^2)\sigma^2 h}{2E_f}$$

where the function g (α,β) depends on the Dundurs parameters [18]. The two Dundurs parameters are the function of the material parameters, tensile and shear moduli, substrate and film. The main point in utilizing equation 1 is to demonstrate also the role of the thickness concerning the structural stability evaluation. Considering the thermal stress equation and the stress raise due to thermal expansion coefficients difference results in:

$$h_c = \frac{G' E_f}{Z\sigma^2}$$

Where, G' is the fracture resistance of the critical thickness at the crack initiation, and Z is a dimensionless driving force number. The comprehensive engagement with the functional aspects of the mechanical response revealed the conflicting pattern of simultaneously achieving high strength and toughness values with relatively more progress to the improvement of the fatigue resistance. Regarding fatigue, theoretical difficulties in unloading are well recognized resulting in phenomenological/empirical working functions. In structural materials a striking example is probably the maraging steel exploration representing a combined product of physical and alloy design concepts. Several strategies have been proposed but the practical translation of global strategies to ultra fine scale segments are still in the category of an ongoing challenge. In more of a global approach for higher strength performance complex solutions have been outlined, e.g. nano-structured materials containing second phase or laminar structures in nano-metallic matrix [19]. In addition, for improved fatigue resistance extrinsic crack-tip shielding mechanisms are often proposed. Thin films of silicon and silicon-based materials are already utilized in MEMS devices and other safety performance applications. The use of single-crystal and polycrystalline silicon thin films extensively in microelectro mechanical systems, required special attention to their mechanical response. Silicon selection was largely based on the relatively high strength and the traditional processing knowledge that have been gained in micro elements techniques. Experimental findings in nano single-crystal silicon particles under cyclic load at ambient temperature identified the following sequential events [17]. First, dislocations nucleate and work hardening occurred with a significant back stress. Second, scale effects were manifested including the fatigue response to be size dependent. In the bulk, silicon has been characterised to be a semi-brittle system with highly hard Ductile-Brittle transition behavior and dislocations activity are not expected at ambient temperature. This argumentation must at the most be deemed in the nano scale regime or in a generalization sense. Dislocation activity by itself is alluding to possible susceptibility to cyclic loading. In fact, the role of cumulative damage was experimentally confirmed. Kahn et al. [20] and Komai et al. [21] have addressed the premature failure in silicon-based films by fatigue. Moreover, under cyclic load the super-hard nano-silicon particles demonstrated significant hysteresis pattern. In this context, the displacement partition between reversible and irreversible strain could be established quantitatively. In some cases the load-displacement curve enabled to track a staircase yield excursion injection. The aforementioned evidence and more resulted in controversial approaches regarding the fatigue process, micro-mechanisms in silicon and silicon-based systems. By excluding the role of dislocations Mulstein et al. [22-24] addressed the cyclic behaviour of a 2 μm thin structural film of polycrystalline silicon. Here, the proposed model centred on the susceptibility of the native oxide layer to the moisture interaction induced cracking. Following this view comparative study was performed with the addition of alkenes-based coating emphasising in small volume segments the surface reaction layer effect. In fact, the susceptibility to stress corrosion in silicon thin films due to humidity interaction has been shown in early work of Komai et al. [25]. Back to the dislocations activity models [17] and the intensive study of silicon particles confirmed the important role of dislocation and twinning activity prior to the failure event. It raised also the alternative possible model of oxide breakage due to sufficient dislocations pile-up underneath the oxide. Thus, the life accounts to the oxide failure and possible growth cycles due to delamination or interface growth. The current author opinion at this stage adopts the conservative approach as the more appropriate for design assessment in small volume performance.

Table. 2. Models comparison

			Energetic	Geometric
Variables	Manson Coffin	Mura et al. [8]	Harvey et al. [10]	Current model
$\Delta\varepsilon_P$ plastic strain amplitude	Dec	Dec	Dec	Dec
τ_f, σ_{YS} the friction and the yield stress	X	X	Dec	Dec
E, μ the elastic moduli	X	Inc	Dec	Dec
γ_S the surface energy	X	Inc	X	Inc

4. Summary

The current investigation emphasizes insights to be addressed by considering the dramatic developments of small volume structures. The study stressed the fatigue response by the assistance of experimental strain control fatigue utilizing novel techniques. Investigation that is centred also on the micro-crack initiation stage affected by residual stresses is supplemented. Beside the practical gain in heteroepitaxial films that became important in devices assisted by layers, the heteroepitaxial growth process provided interesting explorations with respect to the thickness criteria. During heteroepitaxial growth lattice mismatch between the substrate and the film on top of thermal mismatch effects must be considered. The origins of cyclic displacements either mechanical or thermal are insignificant. Thus, thermal expansion and contraction might lead to crack initiation or delamination. Structure integrity requirement also in small volume application remains essential and dislocation activity including high density of threading dislocation might trigger high-density arrays of micro-crack. The aforementioned systems also raised the determination of the critical crack thickness, namely, the thickness beyond which crack formation is favoured [12,13]. In addition, the experimentally determined critical thickness indicated slightly higher values as compared to the model but consistent with the strain energy release rate formulation. Here, it seems appropriate to acknowledge the achievements in terms of mechanical properties due to near-surface nano-layers contribution to upgrade of macro-specimens properties, that could easily be measured [19]. This methodology might be developed also in the nano-scale segments affecting the cyclic crack initiation stage. Following new strategies in engineering materials resulting from optimization of strength and toughness physics due to new concepts. It starts from activities in improving tensile behavior after applying synthesized nano-structured surface layer on bulk metallic materials. This process was studied in copper, alloys, stainless steels indicating remarkable effects as related to plasticity. Finally, activities regarding the standardization of micro materials evaluations that is associated with international framework is currently extensive including also the fracture and fatigue processes.

5. Conclusions

1. In contrast to advances regarding nano-scale mechanical behavior under monotonic loading, fatigue properties achievements have been limited.
2. In fatigue, a conservative design approach is guided mainly by explorations of the complex crack initiation stage. This applies also to intensive efforts in nano-scale multi-layered elastic-plastic systems.
3. Thus, from a technological and design point of view, the fatigue crack initiation stage become a significant first order assessment pillar, at least as a first order assessment methodology.
4. Experimental fatigue activities in small volume segments emphasize the critical role of the local strain amplitude on the initiation life. In small volume cases the domination of the local strain is accentuated.
5. Compressive residual stresses affect the total life. However, experimentally based the findings confirmed the initiation controlled fatigue cases.
6. Considerations regarding the critical thickness have been elaborated, including the analogy to the dislocation dynamic behavior.
7. In silicon-based materials, their MEMS data confirmed that longer fatigue life is mainly connected to the extremely low displacement amplitude interval, with minor evidences to the crack tolerance regime.
8. The critical role of dislocations dynamic in small volume silicon particles has been manifested. The role of stress corrosion processes is only a mechanism that might operate beside other alternatives.
9. The achievement of reliable technological margins on top of processing methodologies improvement as related to nano-scale fatigue properties remains challenging. This long-term goal, promises striking technological potential in various innovation fields.

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